A Study of the Iron-Nickel Santa Catharina Meteorite Using X-PEEM And SEM Techniques.

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INTRODUCTION

The Santa Catharina meteorite is unusual in that it has previously been reported to contain an oxide, possibly spinel, phase within it's structure[1]. As the meteorite is metallic (rather than stoney), it is probable that it originated from within the core of an object of planetary mass that was forming in the early stage of the solar system. Determination of the oxide phase is important as it should provide information on conditions it experienced during it's formation.

Previous work on other fragments of this meteorite (including another piece of this particular sample) have been conducted using electron microscopy techniques such as SEM (scanning electron microscopy), TEM (transmission electron microscopy), EELS, electron diffraction and the like. Whilst these techniques can provide excellent micrographs and information on the crystal structure and stoichiometry, it cannot provide the chemical state information available through x-ray absorption (XAS) techniques. Thus although the ratios of iron, nickel and oxygen can be measured by conventional electron microscopy, how the elements are chemically bonded together is not so straight forward to determine.

It has been demonstrated[2] that XANES (x-ray absorption, near edge structure) measurements at the L-edges of the $3d$ transition metals is a potentially powerful probe of valency and site occupancy. The development of X-ray PhotoEmission Electron Microscopy (X-PEEM) will allow this technique to be applied on structures exhibiting microgrowths of different phases such as this meteorite.

The newly commissioned PEEM2 beamline (7.3.1) at the ALS has been used in ana attempt to determine the oxide phase present in the Santa Catharina meteorite. The data gathered using this technique is being combined with more conventional electron microscopy conducted using the SEM at Southampton Oceanography Centre in the UK.

RESULTS

Two key types of area within the Santa Catharina meteorite have been investigated. The first is a phosphorous rich grain which has apparently led to some form of condensation of nickel within the iron-nickel bulk of the meteorite. The second type of feature we have looked at are the 'islands' containing oxygen which occur in bands across the sample. Analysis of the latter is proving much more complex and is still ongoing, so our comments on these will be brief.

The phosphorous rich grain is illustrated in Figure 1 which shows a comparison between

XPEEM images taken at the iron & nickel L_3 lines and that obtained from the SEM in backscattered mode of the same area. It should be noted that the energy calibration of the 7.3.1 monochromator was not completely correct at the time these data were taken and as yet we have not made the appropriate adjustments. Inset in Figure 1 is an outline sketch illustrating three key regions, A is the 'host' material and is comprised solely of iron & nickel, B is the boundary region, presumably left after condensation of the phosphorous rich grain and is almost devoid of nickel. Region C is the grain itself which contains a much higher nickel / iron ratio than that of the 'host' meteorite. SEM analysis of these three regions indicate a high phosphorous content in region C and no measurable quantities in A or B.
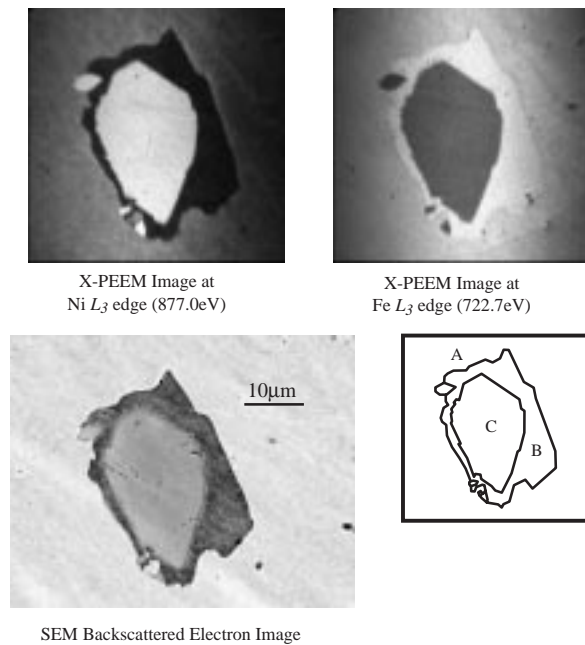

X-PEEM Image at
Ni L_3 edge (877.0eV)

X-PEEM Image at
Fe L_3 edge (722.7eV)

10μm

SEM Backscattered Electron Image

Figure 1: XPEEM images of phosphorous rich grain at Fe & Ni L_3 resonances compared to SEM micrograph of the same area.

Comparison of the sharpness of features in the XPEEM and SEM images indicates a difference depending upon the physical structure in the top layers. A well defined boundary in the SEM image between regions A and B show that the SEM is operating with an overall higher spatial resolution than the PEEM2 instrument (although the latter was not fully optimised for these images). However the relative lack of clarity in the boundary between regions B & C in the SEM image can be attributed to the higher probe depth of the electron beam compared to the X-ray beam. The former being of much higher energy (typically 20kV) will penetrate upto a micron into the surface, revealing structure under the surface layer - in this case the 'shelving' of the grain. The X-PEEM technique in comparison is much more surface sensitive.

Deep penetration of the electron probe in the SEM also gives a relatively large probe size for elemental analysis using the SEM. The XPEEM technique by comparison can produce elemental maps very rapidly at the instruments normal operating spatial resolution.

An unexpected find is of magnetic domains in the almost pure iron boundary region (region B). This is illustrated in Figure 2 which shows the contrast between the maximum of the Fe L_3 peak (the Fe^{2+} resonance at 721.5eV) and offset from this at 722.7eV (where the Fe^{3+} peak would be expected to be). The effect is to highlight the difference between the two domains. Figure 2 also shows the iron L-edge spectra for two of the larger domains, normalised to their pre-edge background levels and the difference spectra of these.

Our investigation of the oxide bearing phases is still ongoing. Three different regions have been investigated using XPEEM and SEM and all present a similar picture to each other. One compli-
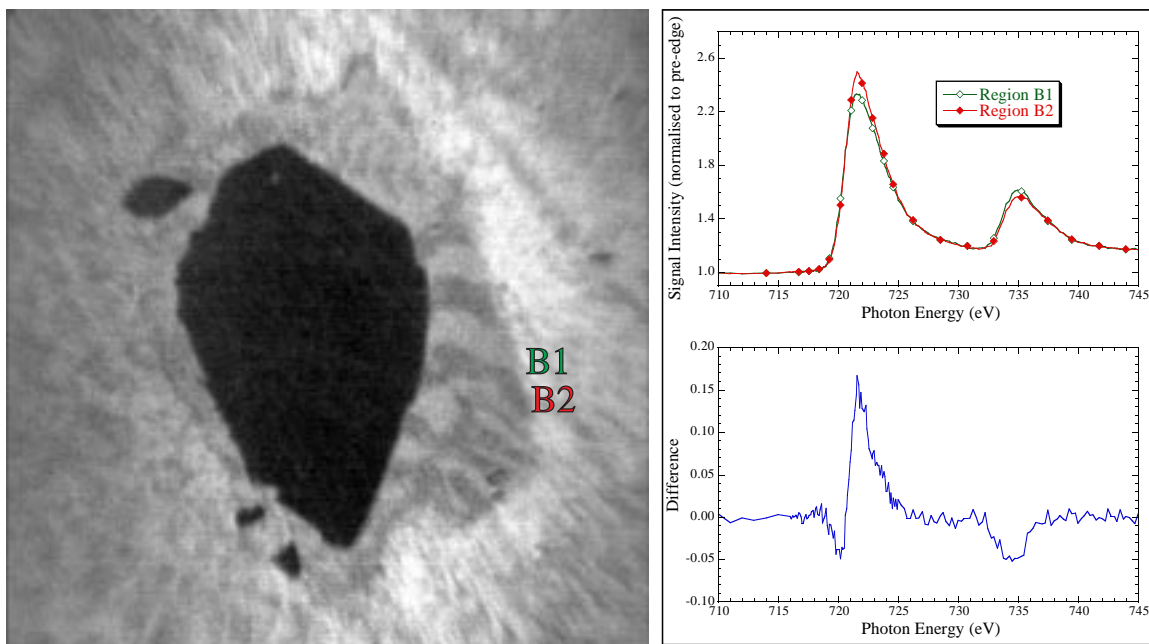
Figure 2 : MCD Image and associated spectra of magnetic domains in boundary region of phosphorous rich grain obtained with PEEM2. Region B1 is one of the large bright bands in the boundary region to the immediate right of the almost black phosphorous rich grain. Region B2 is an adjoining dark band.

cation produced by XPEEM is due to the surface sensitivity. In order to ensure that we examined bulk oxides, rather than surface oxides of the cut face due to exposure to air, polishing lubricants *etc*. it was necessary to ion etch the sample in the PEEM2 sample preparation chamber. This was evidently successful at removing not only the oxygen at the surface, but also from within the top atomic layers of the sample. The deeper probe depth of the SEM has shown that oxide in concentrated in small islands (typically 20 microns in size) and is not evident in the main bulk of the meteorite. Iron *L*-edge spectra collected from these islands using PEEM2 shows an Fe^{3+} resonance in addition to Fe^{2+}. This Fe^{3+} peak is not present in the iron-nickel host. The prescence of Fe^{3+} is a strong indicator of an iron oxide phase.[2]

Whether or not the oxide phases in Santa Catharina are spinels it's hard to say at present. We need to complete our analysis of the data we have and compare it to other XAS measurements we have obtained from model mineral samples. However results obtained so far look promising.

REFERENCES

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